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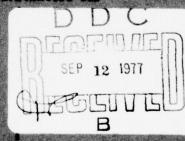
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TECHNICAL REPORT NATICE/TR-77/028



EDIBLE COATINGS FOR INDIVIDUAL FROZEN MEAT PORTIONS

Project Reference: 8:2 177627246H0000-052



ACCUST 1977



Conditionaling Laboratory

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different sources each contributing different attributes to the final coating. No coating examined in this investigation showed any significant improvement over a simple water glaze coating. The study was discontinued due to lack of sufficient funding.

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PREFACE

The storage of meat in the military feeding system often presents certain problems not encountered in the commercial market. One of these is a longer storage period which can lead to moisture loss and oxidative changes deleterious to the product. This work, performed under project number 1T762724AH99BB052 was an attempt to prolong the storage life of frozen meat portions by the application of an edible coating.

The authors wish to thank the members of the Analytical Chemistry Branch of the Food Science Laboratory for their cooperation in providing the analytical data cited in this report. The assistance of Mr. Donald McKee of D. H. McKee, Inc. was also valuable in the testing of the alginate-calcuim chloride coating.

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EDIBLE COATINGS FOR INDIVIDUAL FROZEN

MEAT PORTIONS

INTRODUCTION

Research on edible coatings for meat portions was prompted by the need to provide protection from moisture loss and undesirable oxidative changes during prolonged periods of frozen storage. The criteria for such a coating were:

- 1. The coating would not require removal prior to cooking.
- 2. The coating would be odorless and tasteless.
- 3. The coating would be transparent or translucent.
- 4. The coating would protect the meat from freezer burn, using a minimum of supplemental packaging, for one year at -18° C.
- 5. The coating process must be economically feasible and practical. Therefore the cost should not be higher than other packaging alternatives.

Literature Review

A review was made of current literature to discover any recent innovations in the application of edible coatings. The most promising reference was found in an article by McCormick (1975) who described "Flavor tex" as an alginate-calcium chloride two-stage patented process. It was claimed that this coating would provide an oxygen barrier, a good moisture barrier, and would add some structural identity to the material it encapsulates. Flavor-tex was also described as being a clear, tasteless, odorless film with good binding properties that would not be noticeable after cooking. It is being used on frozen fish portions "minimizing the oxidation effects and doubling the shelf life." Other uses for Flavor-tex include breaded products where it not only protects the food (fish, shrimp, onion rings, etc.), but also prevents moisture migration into the breading. Spraying or dipping may be used to apply each of the two steps in the Flavor-tex process.

Daniels (1973)² reviews 118 patented based processes for edible coatings. Chapter 5, "Meat and Fish Coatings" describes various coatings including the Flavor-tex U.S. patent 3,395,024 awarded to R. Earle³ in 1968. Other patents of interest include the use of glycerol and water; oil-water emulsions using a

McCormick, R.D. 1975. Edible coating isolates oxygen and moisture - seals in flavor. Food Prod Devel. May 1975, pp 14-16.

Daniels, R. 1973. Edible coatings and soluble packaging 1973. Noyes Data Corporation Park Ridge, NJ, 360 pp.

³ Earle, R.D. 1968. Method of preserving food by coating same. U.S. Patent 3,395,024.

low dextose equivalent starch hydrolysate; oil-water emulsions using an edible gum in the water phase; methylcellulose; and combination of paraffinic or olefinic carboxylic acids. Several other coatings found in the patent literature require removal prior to cooking, if the food is not to be cooked in a liquid medium. Lawrence (1973) summarizes the literature on gums and includes their use as coating materials.

In other literature, acetylated monoglycerides, amylose, ethylcellulose and other starch films are described as successful edible coatings. The bibliography, found on page 22, contains not only a list of references cited in this report, but also other literature and patents pertinent to the field of edible coatings.

Personal Communications

Representatives from companies that manufacture products that might be useful as edible coatings for meat were contacted. Among the companies contacted were: D.H. McKee, Inc. (holders of the patent rights for Flavor-tex), Stauffer Chemical Company; Hercules, Inc.; PVO International, Inc.; American Maize Products Company; Atlantic Gelatin Div of General Foods; Stokeley-Van Camp, Inc.; Glidden Durkee Div. of SCM Corp.; Stein-Hall, Inc.; Eastman Chemical Products Inc.; Dow Chemical Co.; W.R. Grace Co.; and Kelco Company. Table 1 lists the companies and addresses contacted.

Varying suggestions were received from the persons contacted. Some felt that the alginate-calcium chloride coating was quite successful; others had reservations about its efficacy. Similar comments were made about acetylated monoglycerides. It was suggested that coatings which require removal prior to cooking might be used in conjunction with enzymes that would dissolve the coating upon reaching a certain temperature. However, it was felt that this approach would be impractical and subject to problems if the meat defrosted at any point. Other possibilities of coating materials were discussed and samples of possible coating materials were received at NARADCOM and screened to determine their potential as a meat coating. Mr. D.H. McKee of D.H. McKee, Inc. demonstrated the use of Flavor-tex in the Food Engineering Laboratory at NARADCOM.

Lawrence, A.A. 1973. Edible gums and related substances. Noyes Data Corporation, Park Ridge, N.J. 342pp.

MATERIALS AND METHODS

The various potential coatings were screened in the laboratory by dipping meat portions in the coating medium. For this study, flaked and formed lamb portions held at -3°C were used. Portions were dipped in the coatings, allowed to dry slightly either by air-drying or with a brief jet of compressed air, then packed between waxed parchment paper, and blast frozen at -29°C. They were removed from frozen storage after 24 hours, examined visually and if promising, grilled and informally examined for off-odors and off-flavors. Those coatings which were considered promising produced a transparent or transluscent film which evenly covered the meat portions. Those screened included Flavor-tex, the calcium chloride, alginate combination distributed by D.H. McKee, Inc.; Durkee 500, a high stability oil, and Paramount C, a hard butter, from Glidden-Durkee Div. of SCM COrporation, Myvacet 9-40, a distilled acetylated monoglyceride, and Myverol 18-98, a distilled monoglyceride, from Eastman Chemical Products, Inc.; Carastay 60, a kappa type, and Carastay 90, an iota type calcium carrageenan, made by Stauffer Chemical Company; Wecotop, a speciality fat, and Dremulse 200-E, a glycerol monostearate, from PVO International; Ediflex, a free-standing amylose film, from American Maize Co; Hallmark K-Dex 4484, a tapioca dextrine, and Hallmark S.D. 3850, a tapioca based food starch, from Stein-Hall, Inc. were received but not examined because these require removal prior to cooking.

Von Ruden (1968)⁵ examined the following products: Klucel, a hydroxypropyl cellulose from Hercules Inc.; Fro-dex, dry corn syrup solids from American Maize Products Co.; Myverol 18-98 (also examined in 1976), Myvacet Type: S, Myvacet 7-00, and Myvacet 5-07, distilled acetylated monoglycerides from Eastman Chemical Co.; and Gelcote a gelatin propylene glycol combination from Atlantic Gelatin Div., General Foods. With the exception of Myverol 18-98, none of these products looked promising enough to examine further.

A summary of the results of the screening examinations done both in 1968 and in 1976 can be found in Table 2. The only coatings that showed promise were Flavor-tex and the two Carastay coatings. A combination of Carastay 60 and 90 appeared to give better coverage than either carrageenan alone. Both the Flavor-tex and Carastay products gave a continuous, somewhat shiny coating and had no detectable off flavor or odors. More work was done to perfect the dipping technique to evenly and consistently coat the meat portions with these products. A problem with the Flavor-tex sticking to either parchment, waxed paper or other meat portions was alleviated by a final cold water dip that further set the calcium alginate coating.

Von Ruden, K.L. 1968. Unpublished data.

Storage Studies

Limited storage studies were undertaken using flaked and formed beef portions with the following variables.

- 1. Uncoated beef portions
- 2. Beef portions with approximately 10 percent alginate-calcium chloride (Flavor-tex) coating.
- 3. Beef portions with approximately 10 percent carrageenan coating (combination of Carastay 60 and 90, kappa and iota types).
 - 4. Beef portions with a comparable (about 10%)water glaze coating.

Samples were placed on waxed parchment paper in one layer in a one-inch deep telescopic box and stored at -12°C and -3°C . The samples at -3°C were analyzed initially and once a week for lipid oxidation (TBA test) and percent moisture over an eight-week period. Appearance and odor of the raw meat portions were also noted weekly and photographs were taken after one month of storage. The samples stored at -12°C were examined initially and monthly over a five-month period for TBA values, percent moisture, odor, and visual appearance. Photographs were taken after one month and five months.

RESULTS AND DISCUSSION

The results of the storage studies, Tables 3 and 4, indicate that any coating used, even a simple water glaze, is beneficial in protecting meat against moisture loss during frozen storage. However, the Flavor-tex samples did not show any marked improvement over the samples with a water glaze coating. From the chemical and visual tests, the Carastay (carrageenam) samples were, in general, slightly superior to all other coated beef samples, but the difference was not considered large enough to warrant its use.

A major difficulty in evaluating any coating is posed by the nature of the coating. Those used were not free standing films. Therefore traditional methods of moisture vapor transfer are almost impossible to carry out. One of the major chemical companies attempted to carry out tests on various gums, but had so many mechanical difficulties in measuring water vapor transmission that tests were discontinued.

The validity of the thiobarbituric acid test (TBA) for lipid oxidation in these products is questionable. The initial TBA values ranged from 1.36 to 3.96 which were relatively high while the TBA values during storage fluctuated greatly.

After five months at -12°C four samples of each variable (uncoated, water glaze, carrengeenan, and Flavor-tex) were analyzed for TBA values. An analysis of variance showed a greater difference within groups than among groups, therefore giving no significant difference in these TBA values. Although the percentages of coating varied somewhat, the differences in percentage coating did not correlate with the differences in TBA values. As shown in tables 3 and 4 the TBA values dropped after the initial analyses, remained relatively level (although with some fluctuating values) until six weeks at -3° C and five months at -12° C, when they increased significantly. This rise in TBA values was greater in the samples stored at -3° C than in those stored at -12° C. Despite the fluctuation, the tests as a whole gave slightly lower TBA values for the carrageenan coated samples stored at both -3°C and -12°C. Few consistent differences were found between the water glaze and Flavor-tex coated samples. The moisture tests are also difficult to assess because moisture loss can come from the outside coating, the meat itself, or, more likely, a combination of both. At -3°C the carrageenan samples retained the most moisture, while at -12°C the results were somewhat inconclusive, showing mainly that the uncoated samples retained the least amount of moisture. At five months at -12°C all of the coated samples had approximately the same percentage of moisture, but results from one to four months indicated that the water glaze and Carastay samples retained more moisture than the Flavor-tex samples.

Visually, the changes in the meat samples were very dramatic. After one week at -3° C the uncoated meat samples had severe freezer burn and continued to deteriorate until the samples were discarded at eight weeks. Also, after only one week at -3° C the Flavor-tex and water glaze samples had moderate freezer burn while

the Carastay samples had very little. After one month at -3° C the water glaze and Flavor-tex samples had severe freezer burn and the Flavor-tex samples also had a whitish appearance from the coating. At this time the Carastay samples had slight to moderate freezer burn but were markedly superior to the other samples. However, after the four-week time the Carastay samples deteriorated in appearance until at seven weeks time all had severe freezer burn.

When stored at -12°C the uncoated samples had severe freezer burn after one month while all the coated samples had little, if any, freezer burn. At three months the coated samples had very slight freezer burn on the top of the samples, but severe freezer burn on the bottom. These observations lead to speculation that the freezer might have reached higher temperatures sometime between the two and three-month period. At the end of five months, when the tests were discontinued, the uncoated samples had severe freezer burn while the coated samples had slight or very slight freezer burn on the top side and severe on the bottom side. The Carastay samples looked only slightly better than the water glaze or Flavor-tex samples. Color comparisons of the samples taken after one month at -3°C and -12°C and 2 months at -12°C showed very clearly the differences in appearance.

Evaluating the samples for off odors gave additional information on the storage stability of the coated and uncoated samples. At -3° C odors indicating slight spoilage were noted in the uncoated samples after four weeks and after six weeks in all the coated samples. In the -12° C samples a very slight objectionable odor was detected after 2 months in the uncoated and Flavor-tex samples, after 3 months in the water glazed samples, and after 4 months in the Carastay samples. The odor was similar to that caused by microbiological spoilage rather than the odor of lipid oxidation. Microbiological tests were not conducted but are important in the study of edible coatings, because two different problems must be considered. One is the ability of the coating to inhibit bacterial penetration into the meat, and the other is to ensure the coating allows enough oxygen permeability to prohibit the growth of anaerobic bacteria. Tables 3 and 4 summarize the results of the chemical, visual, and olfactory results and observations.

The storage studies undertaken on a limited scale reveal some common problems found in chemically evaluating stored meat items, one of which is the lack of a reliable method of determining fat oxidation. Visual examination showed evidence of freezer burn much more quickly and obviously than either the moisture or the TBA tests. The use of the -3° C temperature for accelerated freezer storage tests is worthy of further study. This investigation indicated that the tests of samples stored at this temperature followed the same trends as samples stored at the more commonly used -12° C. Additional tests comparing higher freezer temperatures such as -3° C with the conventionally used storage temperatures of -12° C, -18° C and -24° C, may provide a more rapid means of carrying out accelerated freezer storage tests for screening purposes.

CONCLUSIONS

From this study it can be concluded that the Flavor-tex process does not give a coating that is any more protective against freezer burn than a simple water glaze. The Carastay combinations of iota and kappa carrageenans may offer slightly more protection, but the difference does not seem great enough to warrant its use. It can be theorized that the claims for Flavor-tex's protection hold mostly under refrigerated temperatures or during freezer-thaw cycles when water would not add any protection. One company using the Flavor-tex process on frozen fish blocks did so originally to bind the fish together, and the oxidative and moisture loss protection was an added benefit. The same binding or sticking quality that was a liability in storing individual meat portions would be a benefit to this type of fish processing and also would benefit frozen breaded products. Flavor-tex might offer unique advantages in frozen breaded products by increasing the adhersion of the breading and preventing the migration of water into the breading, thus producing a crisper more adhesive breading.

To develop an edible coating for meat portions would require much more developmental work. In addition to the evaluation of single compounds, the effects of combinations should be explored. By combining compounds one hopefully could utilize the advantages of each, while minimizing the disadvantages.

The carrageenan and alginate gums were promsiing in that they were odorless and tasteless. Other gums might be more protective of the meat than those examined. The gums could also be used as a carrier of compounds which are known to be more effective oxygen and moisture barriers. It has been reported that lipids provide the best moisture barrier, while proteins are effective as oxygen and fat barriers. Oil water emulsions should be tried. Many products that reportedly crack at freezer temperatures, such as amylose esters which are water resistant, soy proteinates, and the acetylated glycerides may be helped by the addition of plasticizers. Glycerine has been mentioned as a plasticizer that helps lower oxygen permeability, and propylene glycol has been used, although care has to be taken to avoid a bitter taste. Polyglycerol esters represent a large number of compounds that reportedly may have good film properties. Gelatin, in combination with other compounds, such as the polyglycerol esters, might be effective. Gelatin had been unacceptable when used with propylene glycol because of bitterness, and gelatin with glycerine reportedly gives a acrid odor upon cooking. Methylcellulose is known to be beneficial in coating materials, and is a possibility in water-oil emulsions. Also, often mentioned is ethylcellulose. However, the present ethylcellulose coatings have to be removed prior to cooking, as do many other water soluble compounds which are suitable only for foods that are cooked in water. Laminates of different compounds processing different protective properties are reportedly effective although they have not been successfully applied to frozen foods without cracking. To avoid discoloration and overheating of the frozen food product any compounds used should be dipped or sprayed at or below room temperature. erature.

Formulating a coating to protect meat or fish portions during frozen storage appears to be feasible. However this development would most likely consist of a system of compounds that would take advantage of the best properties of various products. There remains much developmental work to be done in the field of edible coatings to produce a product that would be economically feasible in protecting meat items from freezer burn and oxidative rancidity. However, work on this project has been discontinued due to lack of funding.

Table 1. Companies contacted for information on edible coatings

N	a	me

American Maize Products Co.

Atlantic Gelatin Div., General Foods

D. H. McKee, Inc.

Dow Chemical Co.

Gidden Durkee Div., SCM Corp.

Eastman Chemical Products Inc.

Hercules, Inc.

Kelco Co.

PVO International, Inc.

Stauffer Chemical Co.

Stein Hall, Inc.

Stokely Van Camp, Inc.

W.R. Grace Co.

Address

250 Park Ave. New York, New York 10017

Hill Street Woburn, Mass 01801

P. O. Box 10192 Tampa, Florida 33609

2040 Dow Center Midland, Michigan 48640

900 Union Commerce Building Cleveland, Ohio 44115

Kingsport, Tenn 37622

F & FD Div Wilmington, Del 19899

8355 Aero Drive San Diego, Calif 92123

Chemical Specialities Div. P. O. Box 2167 Morristown, New Jersey 07960

Food Ingredients Div. Westport, Conn 06880

605 Third Ave. New York, New York 10016

Central Laboratories 6815 East 34th St. Indianapolis, Ind. 46226

Cryovac Div. Spartanburg, S.C. 29302

Table 2. Results of Screening Edible Coating Compounds

	lable 2. Results of Screening Edible Coating Compounds	ore coaring compounds	
Product	Company	Composition	Comments
Carastay 60	Stauffer Chem Co.	Carrageean gum	Colorless, odorless, tasteless coating
Carastay 90	Stauffer Chem Co.	Carrageean gum	Colorless, odorless, tasteless, coating
Durkee 500	Gidden Durkee Div., SCM Corp	High stability oil	Opaque coating on freezing
Drewmulse 200-E	PVO International, Inc.	Glycerol monostearate	Opaque coating on freezing
Ediflex	American Maize Products Co.	Amylose film	Received as film samples, sl. sweet oily & bitter tasting. Did not dissolve on grilling and became more bitter
Flavor-tex	D.H. McKee, Inc.	Alginate-Calcium Chloride	Gave an odorless, tasteless, colorless film
Frodex*	American Maize Products Co.	Dry corn syrup solids	Sweet flavor, very thin solution, gave sticky coating
Gelcote*	Atlantic Gelatin Div, General Foods	Gelatin, propylene glycol	Bitter flavor
Klucel*	Hercules, Inc.	Hydroxypropyl cellulose	Very sticky coating
Myvacet 5-07*	Eastman Chem products Co.	Distilled acetylated monoglyceride	Applied hot; discolored meat, cracking on freezing
Myvacet 7-00*	Eastman Chem Products Co.	Distilled acetylated monoglyceride	Applied hot; discolored meat sl. cracking on freezing
Myvacet Type S*	Eastman Chem Products Co.	Distilled acetylated monoglyceride	Applied hot; discolored meat severe cracking
Myvacet 9-40	Eastman Chem Products Co.	Distilled acetylated monoglyceride	Applied hot; discolored meat

Table 2 (Continued)

Comments	Applied hot, discolored meat	Opaque coating	Opaque coating
Composition	Distilled monoglyceride	Hard butter	Speciality fat
Company	Eastman Chem Products Co. Distilled monoglyceride	Gidden Durkee Div SCM Corp Hard butter	PVO International, Inc.
Product	Myverol 18-98*	Paramount C	Wecotop

*examined in 1968 by K.L. Von Ruden

Table 3. Storage Studies of Beef Portions With Various Coatings $(-3^{\circ}\mathbb{C})$

	2014	Joseph 1	C. Mooke	o look	odeout II	o look	S Mooke	The state of	
	Initial	меек	∨ weeks	3 weeks	4 weeks) weeks		weeks	o weeks
Uncoated TBA	3.96	1.40	1.45	1.31	1.96	1.81	6.29	10.37	15.64
Moisture	65.65	62.99	26.49	62.74	62.74	62.74	63.44	61.24	57.74
Appearance	typical	severe freezer	severe freezer	severe freezer burn	severe freezer	severe freezer burn	severe freezer burn	severe freezer burn	severe freezer burn
Odor	OK	OK	OK	OK	v. sl. spoiled	v. sl. spoiled	sl. spoiled		sl. spoiled mod. spoiled
Water Glaze	1.33	1.64	0.85	0.62	1.86	2.05	9.36	12.90	10.63
Total Moisture	e 72.38	69.84	69.12	67.93	65.76	65.31	59.92	56.27	59.20
% Coating*		11.0	12.4	12.9	11.9	10.7	11.5	9.3	8.6
Appearance	glossy	рош	рош	рош	pou	рош	severe	severe	severe
	ice crystals	freezer	freezer	freezer	severe	severe	freezer	freezer	freezer
		pnrn	pnru	purn	freezer	freezer	pnru	pnru	purn
Odor	ОЖ	OK	OK	OK	burn OK	burn OK	sl. spoile	d sl. spoil	sl. spoiled sl. spoiled sl. spoiled
Flavor-tex									
TBA	3.78	1.15	1.12	1.07	2.97	2.66	5.94	9.70	12.93
Total Moisture	9	24.69	67.32	64.95	64.02	62.85	60.93	59.73	58.91
% Coating*	12.0	11.0	12.1	12.7	11.2	9.7	12.4	10.7	8.8
Appearance	glossy-ice	рош	рош	pou	рош	рош	рош	severe	severe
	crystals	freezer	freezer	freezer	freezer	severe	severe	freezer	freezer
		purn	pnru	burn	burn	freezer	freezer burn.	burn	burn
				whitish	whitish	whitish	whitish	whitish	whitish
				grate	10100		10100	10100	10100
*% by weight	*% by weight of coating when originally	n originall	y applied						

18

Table 3 (Continued)

	Initial	1 Week	2 Weeks	3 Weeks	4 Weeks	5 Weeks	6 Weeks	7 Weeks	8 Weeks
Odor		OK N	OK	У	OK	OK Y	v. sl spoiled	v. sl. spoiled	v. sl. spoiled
Carastay	1.36	0.74	0.77	1.15	1.51	1.39	5.02	7.35	9.12
Moisture 71.60	71.60	70.82	69.66	68.97 13.8	67.97	67.75	65.58	65.60	64.77
Visual Appéarance	glossy-ice crystals	v. sl. freezer burn	sl. freezer burn	sl. freezer sl. to sl. to burn mod freezer mod burn freezer	sl. to er mod freezer burn	mod freezer burn	mod to severe freezer burn	severe freezer burn	severe freezer burn
Odor	OK.	Ж	OK	OK	OK	OK	v. sl. spoiled	v. sl. spoiled	v. sl. spoiled

* \$ by weight of coating when originally applied.

Table 4. Storage Studies of Beef Portions with Various Coatings

ed ture ting * rance	Initial 3.12 68.46 typical freezer burn OK 3.86.	1 Month 1.46 64.55 - severe freezer burn 0K	2.90 62.71 severe freezer burn v. sl. spoiled 2.62	3 Months 2.66 63.98 severe freezer burn v. sl. spoiled 1.33	4 Months 2.09 63.02 v. sl. spoiled 1.55	5 Months 3.81** 61.58** severe freezer burn v. sl. spoiled 3.50**
Total Moisture Coating* Visual	71.12 12.1	70.29 12.5	69.93 12.2 v. sl. freezer	69.19 9.8 v. sl. freezer	70.90 13.9	66.70** 10.9** sl. freezer burn
ear and		v. st. freezer	v. st. freezer burn on edges	v. Si. ireczer burns on top severe on bottom	si. Treezer ourn top mod-severe on bottom	on top severe on bottom
Odor Flavor-tex TBA	3.33	OK 1.29	ok 1.60	v. sl. spoiled	v. sl. spoiled	v. sl. spoiled
Moisture % Coating*	69.43 13.6	68.20	67.47	68.18 9.8	67.70	67.20** 9.4**
Appearance	ice crystals glossy	v. sl. freezer burn on edges	v. sl. freezer burn on edges	v. sl. freezer burn on top severe on botto	v. sl. freezer sl. freezer burn burn on top on top edges severe on bottom severe on bottom	sl. freezer burn on top severe on bottom
	OK	OK	v. sl. spoiled	v. sl. spoiled	v. sl. spoiled	v. sl. spoiled

^{* %} by weight of coating when originally applied. ** mean of four samples.

Table 4 (Continued)

Initial	arastay 2.37 TBA 2.37 Total	Moisture 69.78 Coating # 12.7	Appearance ice crystals glossy	odor OK
1 Month	1.10	70.15	is ice crystals glossy no freezer burn	OK
2 Months	0.81	70.17	v. sl. freezer burn on edges	OK
3 Months 4	2.18	71.48	v. sl. freezer v. sl. freezer v. sl. freezer v. sl. freezer burn on top burn on top mod to severe mod to severe	OK
4 Months	1.73	69.70	v. sl. freezer burn on top mod to severe	on bottom v. sl. spoiled
5 Months	3.22**	67.19**	v. sl. freezer v. sl. freeze burn on top burn on top mod to severe mod to severe	on bottom on bottom v. sl. spoiled v. sl. spoiled

^{* %} by weight of coating when originally applied.

^{**} mean of four samples.

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